TRANSESTERIFICATION OF RUBBER SEED OIL USING HETEROGENEOUS CATALYST

MOHD ZAIDI BIN MAMAT

CHEMICAL ENGINEERING
MAY 2013
UNIVERSITI TEKNOLOGI PETRONAS
BANDAR SERI ISKANDAR

31750 TRONOH

PERAK

CERTIFICATION OF APPROVAL

TRANSESTERIFICATION OF RUBBER SEED OIL USING HETEROGENEOUS CATALYST

By

MOHD ZAIDI BIN MAMAT

A project dissertation submitted to the

Chemical Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfillment of the requirement for the

BACHELOR OF ENGINEERING (Hons.)

(CHEMICAL ENGINEERING)

Approved by,

(DR. Bawadi Bin Abdullah)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(MOHD ZAIDI BIN MAMAT)

ACKNOWLEDGMENT

I would like to express my highest gratitude to my supervisor, DR. Bawadi Bin Abdullah who has been patiently helping and guiding me in spite of his very busy schedule to monitor my progress project, provide information and sharing his knowledge throughout the year.

My highest appreciation also goes to Siti Shafriena for her guidance and advice whenever I face difficulties in my project. I also wish to thank the staff in the laboratory for their precious knowledge and supportive spirit as they have always been helping and assisting me on my project tasks.

Not to forget, I would like to thank all my colleagues and family who always support me and last but not least, I would like to thank everyone who has contributed directly or indirectly and gave me moral support and inspiration over this period.

ABSTRACT

Biodiesel is a domestically produced and renewable type of fuel that can be manufactured from edible or non-edible oils. Biodiesel's physical properties are nearly the same to those of petroleum diesel, but it is a cleaner-burning alternative. Biodiesel consist of long-chain alkyl esters which is typically made by chemically reacting lipids with an alcohol producing fatty acid esters.

In this project, biodiesel is produced by transesterification of rubber seed oil using calcium based catalyst. Catalysts of CaO and $CaCO_3$ is prepared by using calcination and CaO/ZnO by incipient wetness impregnation method.

For this project, the transesterification reaction of the rubber seed oil was carried out under the same reaction condition in the presence of calcium carbonate ($CaCO_3$), calcium oxide (CaO), and mixed calcium oxide (CaO/ZnO) catalyst and the result shows that the conventional KOH catalyst had better performance than the developed calcium based catalyst. The biodiesel produced from KOH contain the highest fatty acid methyl ester content, about 80.02% for biodiesel from rubber seed oil and 77.96% for biodiesel from palm oil. Among the synthesized catalyst, the mixed calcium oxide catalyst perform better than the other two catalyst and the $CaCO_3$ catalyst has the lowest performance.

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CHAPTER 1
INTRODUCTION

1.1 Background of study.

Currently, the world's fossil fuel resources are depleting due to continuous consumption and increasing demand of the fuel. To solve the crisis, alternative fuel sources are being developed taking into consideration of greenhouse gas emission due to the excess use of petroleum based fuels leads to the global warming and climate change for example biodiesel, bioalcohol, biomass, biogas and synthetic fuels.

Among the alternatives, biodiesel which is a type of fuel that is produced from either edible or non-edible oil through the transesterification process can be used directly in the diesel engine, while others need some sort of modification before they can be used as substitute of conventional fuels [Morshed, M. (2011)].Various countries are looking for different types of vegetable oils as substitutes for diesel fuels depending on their climate and soil conditions. For example, soybean oil in the US, rapeseed and sunflower oils in Europe, palm oil in South-east Asia and coconut oil in the Philippines are being considered [Morshed, M. (2011)]. But, the production of biodiesel from edible oils will causing a competition between biodiesel feed stock and food supply in the long term[8]. Thus, non-edible oils is now being consider as an alternative to produce the biodiesel instead of edible oil. The use of non-edible oils when compared with edible oils is very significant in developing countries because of the tremendous demand for edible oils as food, and they are far too expensive to be used as fuel at present.

Various methods for the production of biodiesel have been reported such as transesterification, pyrolysis, microemulsification, and direct use and blending method.[4] Among the methods, transesterification is a widely used and attractive technique.The study will be focusing on the transesterification of the rubber seed oil using heterogeneous catalyst.

1.2 Problem Statement.

In the transesterification reaction, homogeneous base or acid catalysts, hydrochloric acid or sulphuric acid, are usually used to prepare the biodiesel[Loreto,E..et.al,2005]. However, a high molar ratio of methanol to oil is needed, and the reaction time is very long [Zhang,Y.,2003]. So, the base catalysts are more preferred to be used because the catalytic activity of a base is higher, short reaction time and relatively low temperature can be used with only a small amount for catalyst and with little or no darkening of colour of the oil [Balat,M.,2011]. However, the drawback of using conventional homogeneous catalyst is that it is difficult to remove these catalysts, and separating and cleaning the catalyst and product consume a lot of water. Thus, the usage of heterogeneous catalyst which are environmentally friendly are more preferred. The replacement of homogeneous catalysts will give various advantages such as the reduction of environment pollutants, easy product purification and also easy catalyst separation from the reaction mixture [Arponcai,S.C, et.al, 2012].

1.3 Objective

The main objectives of this study:

i) To synthesize calcium based catalyst.

ii) To produce biodiesel from rubber seed oil using transesterification reaction.

1.4 Scope of study

The scopes of study based on the objectives are as follow:

i) Conduct acid esterification and transesterification of rubber seed oil.

ii) Analyze and compare the effectiveness of the catalysts used.

1.5 Relevancy of the project

The research on biofuel is important to help reduce the usage of petroleum based fuel and in the future, the biofuel will replace the fossil fuel as the world's fossil fuel supplies keep decreasing day by day. The use of biofuel also will help solving the environmental problem involving the emission of greenhouse gas produced from the combustion of petroleum based fuel.

1.6 Feasibility of the project

The project will be conducted on the usage of heterogeneous catalyst as the catalyzer to the transesterification process of rubber seed oil. The effectiveness of the catalyst is analyze and compared. The project are expected to be completed within time frame as presented in the gantt chart if the experiments conducted does not encounter problems or errors which causing the experiments need to be repeated again. The equipments needed for the experiment and analysis are also already available inside UTP.

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CHAPTER 2
LITERATURE REVIEW

2.1 Rubber Seed Oil.

Rubber seed oil is extracted from the seeds of rubber trees. In the latex manufacturing process, rubber seeds usually are not collected and commercialized. Rubber seeds are an abundant source of non-edible oil that is available in Malaysia. Nonetheless, the seeds remain underutilised although oil produced has the potential to be used in a number of applications.

2.1.1 Advantages of using rubber seed to produce Biodiesel.

There are so many advantages associated with the use of rubber seed to produce Bio-diesel.

1. Rubber seed is a rich source of essential fatty acids which should have made it a great dietary source of nutrients. But it is poisonous, rubber seed contains cyanogenic glycoside which releases hydrogen cyanide in the presence of enzymes.

2. Rubber tree do not necessarily require cultivation of new plantations. Large plantations of Rubber trees meant for the production of rubber latex are abundant in Asia and Africa.

3. It is more profitable to farmers because they can now make extra profit from harvesting rubber seeds.

4. It will re-invogorate the economy of local communities which have abandoned their plantations due to low demand for rubber latex.

5. Rubber seed produces more oil, and exploring existing plantations around the world will go a long way in providing large percentage of world energy consumption.

2.1.2 Fatty acids in rubber seed oil

The fatty acid composition and the important properties of rubber seed oil in comparison with other oils can be seen in Table 1. Rubber seed oil consists of 19.9% saturation fatty acid comprising of palmitic and stearic acids and 80.1% unsaturation fatty acid comprising mainly of oleic, linoleic and linolenic acids. Saturation fatty acid methyl esters increase the cloud point, cetane number and improve stability whereas more polyunsaturates reduce the cloud point and cetane number and stability.

Table 1 : Properties of rubber seed oil in comparison with the other oils (Ramadhas.et.al,2005)

Property	Rubber seed	Sunflower oil	Rapeseed oil	Cotton seed oil	Soybean oil
Fatty acid composition (%)					
(i) Palmitic acid $C_{16:0}$	10.2	6.8	3.49	11.67	11.75
(ii) Stearic acid $C_{18:0}$	9.7	3.26	0.85	0.89	3.15
(iii) Oleic acid $C_{18:1}$	24.6	16.93	64.4	13.27	23.26
(iv) Linoleic acid $C_{18:2}$	39.6	73.73	22.3	57.51	55.53
(v) Linolenic acid $C_{18:3}$	16.3	0	8.23	0	6.31
Specific gravity	0.91	0.918	0.914	0.912	0.92
Viscosity (mm^2/s) at 40°C	66.2	58	39. 5	50	65
Flash point (°C)	198	220	280	210	230
Calorific value (MJ/kg)	37.5	39.5	37.6	39.6	39.6
Acid value	34	0.15	1.14	0.11	0.2

The type and percentage of fatty acids contained in vegetable oil depends on the plant species and on the growth conditions of the plant. Though vegetable oils are of very low volatility in nature, it quickly produces volatile combustible compounds upon heating.

2.2 ACID ESTERIFICATION

Bio-diesel production from normal oil requires esterification reaction of lipid with alcohol in the presence of alkaline catalyst. But the presence of high free fatty acids of the rubber seed oil can discourage it's uses for Biodiesel. Free fatty acids combines with alkaline catalyst and initiate saponification to produce soap which inhibits the separation of glycerol and esters. Hence, another reaction must be initiated to prevent saponification before the actual formation of the diesel. The reaction involves the Fischer Esterification before alkaline-catalysed Trans-esterification.

Acid esterification is simply the purification of the rubber seed oil. It involves a reaction of the oil with alcohol in the presence of an acid catalyst, in this project sulphuric acid are used as catalyst, to convert the free fatty acid contents to monoglycerides thus reducing free fatty acid of the oil to less than 2% which enable the oil to be used in transesterification process.

$$\underset{\text{FFA}}{R-COOH} + \underset{\text{Alcohol}}{R_4OH} \xrightarrow{\text{Catalyst}} \underset{\text{Water}}{H_2O} + \underset{\text{Ester}}{R-CO-O-R_4}$$

Figure 1 : Acid-esterification reaction.

Acid-catalysed esterification has its own problem. The reaction of Alcohol (R'-OH) with FFA (RCOOH) in the presence of an acid (H^+) reversible and at equilibrium. This implies that, we may not be able to obtain the product of the reaction.But 'Le Chatelier principle' shows that, if we increase one of the reactants ,alcohol or oil, or remove one of the products, mono-ester or water, the equilibrium will shift to the right. Hence, it is essential that the amount of the alcohol used should be in excess.[Agbogun,J,2011] The amount of FFA (RCOOH) in the oil in the reaction must not be in excess because there must be enough alcohol to donate the alkyl group (R') to form the mono-esters(RCOOR'). Also, removal of reactant is not recommended because it is better to carry out separation of products at the end of the reaction.

According to A.S Ramadhas, the optimum condition for methanol to oil molar ratio is 6:1. According to Modhar A.Khan, the lowest FFA content in the biodiesel can be obtained at 65°C. For this project, acid-esterification reaction conducted with methanol to oil molar ratio of 15:1, sulphuric acid catalyst concentration of 10% and at a reaction temperature of 65°C.

2.3 TRANSESTERIFICATION

Transesterification is the actual process of formation of the Bio-diesel. Before starting the trans-esterification process, it is essential to take the following precautions:

1. Water must be completely separated from the product formed from acid esterification. Otherwise, water content may initiate saponification.
2. The alcohol must be dry.
3. The alkaline should be Concentrated NaOH or KOH.

Transesterification is the chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters. The long and branched chain triglyceride molecules are transformed to monoesters and glycerine. Transesterification process consists of a sequence of three consecutive reversible reactions. That is, conversion of triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides. The glycerides are converted into glycerol and yielding one ester molecule in each step. The properties of these esters are comparable to that of diesel. The overall transesterification reaction can be represented by the following reaction scheme.

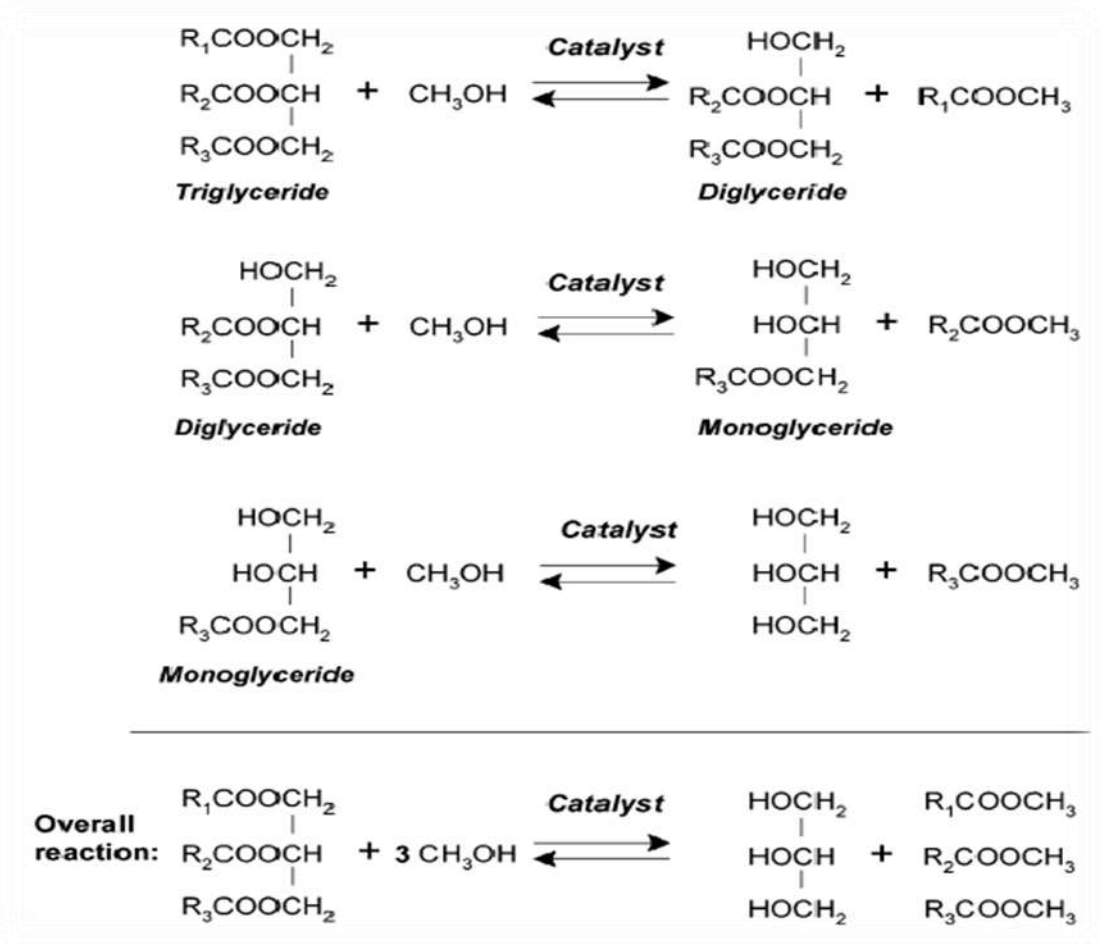

Figure 2 : Transesterification reaction.

Stoichiometrically, three moles of alcohol are required for each mole of triglyceride, but in practice a higher molar ratio is employed in order to displace the equilibrium for getting greater ester production. Though esters are the desired products of the transesterification reactions, glycerine recovery also is important due to its numerous applications in different industrial processes.

Usually, the transesterification reaction used short chain alcohols are methanol, ethanol, propanol and butanol. The yield of esterification is independent of the type of alcohol used. Therefore, the eventual selection of one of these three alcohols will be based on cost and performance considerations.

For this project, transesterification of rubber seed oil are conducted under reaction condition of methanol to oil ratio of 6:1, catalyst concentration of 5 wt% and reaction temperature of 65°C.

2.4 CATALYST

A catalyst is a substance that speeds up a reaction, but it is chemically unchanged at the end of the reaction. The amount of a catalyst used does not change during a reaction because it is not consumed as part of the reaction process. Catalysts lower the energy required to reach the transition state of reaction, allowing more molecular interactions to achieve that state. Although catalysts affect reaction kinetics, the equilibrium state remains unaffected.

There are two kinds of catalysts typical to any biodiesel process: homogeneous and heterogeneous. Homogeneous catalysts function in the same phase as the reactants. Typically, homogeneous catalysts are dissolved in a solvent with the substrates. Heterogeneous catalysis is the opposite of homogeneous catalysis, meaning it occurs in a different phase than the reactants. Most heterogeneous catalysts are solids that act on substrates in a liquid or gaseous reaction mixture.

Generally, transesterification is a catalyzed chemical reaction. Hydroxide or methoxide of sodium or potassium is used as a homogeneous catalyst in this reaction. However, homogeneous catalyst has its disadvantage such as its intolerance of moisture and free fatty acids (FFA) in feedstock, the generation of a substantial amount of process wastewater, and incapable of catalyst reuse, the conception of heterogeneous catalysts evolves. Heterogeneous catalysts shows potential in the transesterification process. The catalyst can be reused (cost effective), it has a tolerance of moisture and FFA which allows the use of lower-quality used/waste oils for the transesterification process, it is inexpensive has low methanol solubility, is non-corrosive and environmental friendly.

For this project, three types of heterogeneous catalyst is synthesized, that is $CaCO_3$, CaO, and CaO/ZnO catalyst.

2.5 ANALYSIS METHOD

The biodiesel that are produced from the transesterification of the rubber seed oil will be analyzed by using Gas Chromatography (GC) with CBBR method. Gas chromatography used in analytic chemistry for analyzing compounds that can be vaporized without decompose it. Typical usage of GC include testing the purity of a particular substance, or separating the different components of a mixture. For this project, the content of the fatty acid methyl ester of the biodiesel is calculated by using the data from the graph of gas chromatography result.

CHAPTER 3
METHODOLOGY

3.1 APPARATUS

retort stand, pipette, 500 ml beaker, 50 ml burette , hot plate with magnetic stirrer, 250 ml 3-necked round bottomed flask with reflux condenser, separating funnel , crucible, drying oven, furnace, water bath, analytical balance.

3.2 CHEMICAL

Rubber seed oil, n-hexane, iso-propanol, phenolphthalein, potassium hydroxide, calcium carbonate, calcium oxide, zinc nitrate hexahydrate, , toluene, methanol, sodium sulphate.

3.3 CATALYST SYNTHESIS.

For the project, three catalyst were synthesized in order to study their potential in the production of biofuel. Another reason to synthesis catalyst is to activate the catalyst for the reaction to produce biofuel.

$CaCO_3$

1. Weight and record the mass of the calcium carbonate.
2. Heat the calcium carbonate in a furnace at 873 K for 3 hours with heating rate of 293 K/min.
3. Take out the calcined calcium carbonate, then weight and record the mass.
4. Store the Calcium oxide inside airtight container.

CaO

1. Weight and record the mass of the calcium carbonate.
2. Heat the calcium carbonate in a oven or furnace at 1173 K for 2 hours with heating rate of 293 K /min.
3. Take out the calcined calcium carbonate, then weight and record the mass.
4. Store the Calcium oxide inside airtight container.

CaO/ ZnO.

1. 5.5g of Zn(NO3)2.6H2O and 28.5g of CaO powder are weighed.
2. Zn(NO3)2.6H2O are dissolve in 80 ml of distilled water.
3. The formed solution was stirred continuously using the magnetic stirrer and CaO are added.
4. The mixture is placed on the hot plate stirrer in water bath and left for 4 hours until it become paste.
5. The heater is on for the last 1 hour at temperature 45℃.
6. The paste is checked for pH value by using the pH meter before entering the oven for drying.
7. Then, the paste is placed in an oven at the temperature of 100°C, and again left for 20 hours for drying.
8. Weight the sample before calcinations process.
9. The sample is calcined at 900℃ for 2 hours.
10. The sample is cooled under room temperature and weight.

3.4 ACID VALUE TEST.

Acid value test was conducted to determine the FFA content of oil sample before and after acid-esterification reaction. Standard method used for this procedure is according to EN 14104.

1. 3g of sample is dissolved in iso-propanol in conical flask.
2. Add phenolphthalein indicator into the flask
3. Titrate solution with a 0.1M of potassium hydroxide (KOH) until the mixture turn light pink.
4. Record the volume of the KOH used.

Acid value can be calculated using formula:

$$AV = \frac{MW \times vol \times N}{m} \qquad (1)$$

Where:

Vol : volume of titrant used, ml

N : normality of KOH, 0.1 mol

m : mass of sample, g

MW : MW of KOH, 56.1g/mol

Free fatty acid can be calculated using formula:

$$FFA = \frac{AV}{f}$$

Where f is conversion value factors for RSO =2.81

3.5 ACID ESTERIFICATION.

1. 50g RSO are heated to its reaction temperature of 338 K in 3-necked round bottom flask with reflux condenser.
2. Methanol are added according to molar ratio of 15:1.
3. 10 wt% of H_2SO_4 catalyst is added and start the reaction time.
4. The reaction mixture is left for 3 hours with stirring rate of 350 rpm.
5. After 3 hours, the reaction was stopped by adding 323 K of distilled water alternately with hexane and left overnight in separating funnel for separation.
6. The bottom layer of the mixture is removed.
7. Add sodium sulphate to absorb excess water.
8. Do acid value test.

3.6 TRANSESTERIFICATION.

1. 50g of RSO are heated to its reaction temperature of 338 K in 3-necked round bottom flask with reflux condenser.
2. Methanol are added according to molar ratio of 5:1.
3. 5 wt% of catalyst is added and start the reaction time.
4. The reaction mixture is left for 3 hours with stirring rate of 350 rpm.
5. After 3 hours, add 40ml of acetic acid to stop the reaction and the products are left overnight to allow the product to separate into two layers.
6. The lower layer, which contained impurities and glycerol, is drawn off.
7. Add 333 K of distilled water and stir the mixture gently and then drawn off the water. This process is repeated until the water at lower layer becomes clear.

8. Add sodium sulphate to absorb excess water and then filter the mixture to get the oil.

3.7 Gas Chromatography sample preparation.

1. Add 0.05g of oil sample into vial and record the exact weight.
2. Add 1 ml of Methyl hepta decanoate into the vial.
3. Send the sample for GC analysis .

3.8 Experimental Flow Chart.

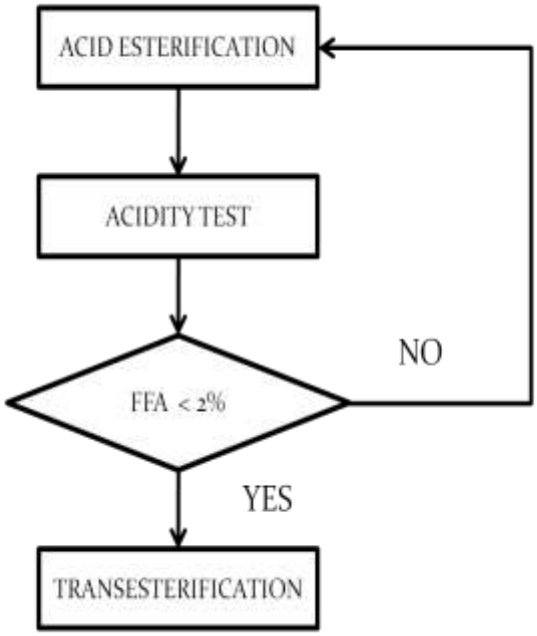

FIGURE 3 : Flow Chart Of Experiment

3.9 GANTT CHART AND KEY MILESTONES.

NO	WEEK / DETAIL	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
Final Year Project I																	
1	Selection of Project Title				●												
2	Preliminary Research Work and Literature Review							●									
3	Extended Proposal Writing							●									
5	Oral Presentation											●					
6	Interim Report Writing														●		
Final Year Project II																	
1	Project Work																
2	Progress report Writing									●							
3	Draft report Writing													●			
4	Dissertation Writing (soft bound)														●		
5	Technical paper Writing														●		
6	Oral Presentation															●	
7	Dissertation Writing (hard bound)																●

● Suggested milestone

Process

CHAPTER 4
RESULTS

4.1 Catalyst calcination.

Calcium oxide catalyst it produced from calcinations of calcium carbonate powder. The mass of the catalyst before and after calcination are recorded.

Table 2 : Calcination Of Calcium Carbonate.

Catalyst	Time (hour)	Temperature (°C)	Weight before calcination (g)	Weight after calcination (g)
CaO	2	900	30.0078	17.1861
$CaCO_3$	3	600	60.1300	59.6000
CaO/ZnO	2	900	30.5023	29.3500

Calcium oxide catalyst obtained from calcination of calcium carbonate at the temperature of 900°C for 2 hours. Upon heating, calcium carbonate undergoes a reaction where bound CO_2 is released to the atmosphere and leave only calcium oxide. The sample weight loss is 12.8217 grams. Theoretically, weight loss occurred from the decomposition of calcium carbonate to calcium oxide is about 44% and from the result, the weight loss is 42.73%.

Calcium carbonate catalyst heated to a temperature of 600°C to activate it.60.13g of CaCO3 were heated and after the calcination, the weight of the catalyst reduced to 59.6g. Theoretically, the weight loss should be 0% but from the result which can be observe from table 2, about 0.88% of the catalyst weight are reduced. This could be due to the catalyst used contain some moisture before it is used for calcination process.

The CaO/ZnO calcined at a temperature of 900°C for 2 hours. From the result there is mass reduction observed.

4.2 Acidity Test.

Acid value test was conducted to determine the FFA content of rubber seed oil sample after acid-esterification reaction. Standard method used for this procedure is according to EN 14104.

Table 4 : Free Fatty Acid Content (before acid-esterification)

Trial	Sample weight (g)	Volume of KOH used (ml)	Fatty acid content (%)
1	1.0200	18.9	36.99
2	1.0230	19.2	37.22
3	1.0100	18.6	36.77
		Average	36.99

Table 5 : Free Fatty Acid Content (after acid-esterification)

Trial	Sample weight (g)	Volume of KOH used (ml)	Fatty acid content (%)
1	1.0110	0.34	0.68
2	1.1000	0.36	0.65
3	1.0718	0.35	0.67
4	1.1320	0.37	0.65
		Average	0.66

For the rubber seed oil to be able to be used in transesterification reaction, its free fatty acid content must be lower than 2%. From the acidity tests result, the percentage of the free fatty acid content is calculated for the oil sample that did and did not go through acid-esterification process. From Table 4, the average free fatty acid content of the rubber seed oil that did not go through the acid esterification reaction is about 37%, which is higher than the maximum allowable free fatty acid

content of 2%. From table 5, the average free fatty acid content of the rubber seed oil that had gone through the acid esterification reaction is 0.66%. This shows that that the acid esterification process had successfully reduced the fatty acid content and the oil can be used in the transesterification process to produce biodiesel.

4.3 Gas Chromatography analysis.

Standard method of EN 14103 is used to verify the ester content of fatty acid methyl esters (FAMEs) between C_{14} and C_{24}. From this analysis, percentage of methyl ester content is obtained with a solution of methyl heptadecanoate as internal standard. The FAME content is calculated using the formula of :

$$C = \frac{\Sigma A - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100\% \qquad (2)$$

Where:

$\sum A$ = total peak area $C_{14:0} - C_{24:1}$

A_{IS} = internal standard (methyl heptadecanoate) peak area

C_{IS} = concentration of the internal standard solution, in mg/mL

V_{IS} = volume of the internal standard solution used, mL

m = mass of the sample, in mg

Table 6 : FAMEs content in biodiesel

Oil Type	catalyst	FAME content (%)
Palm Oil	KOH	77.96
Palm Oil	CaO	59.30
Palm Oil	$CaCO_3$	58.76
Palm Oil	CaO/ZnO	69.44
Rubber Seed Oil	KOH	80.02
Rubber Seed Oil	CaO	66.61
Rubber Seed Oil	$CaCO_3$	63.92
Rubber Seed Oil	CaO/ZnO	75.82

From the gas chromatograph result of transesterification of palm oil and rubber seed oil, the fatty acid methyl esters (FAME) content are calculated and tabulated . All experiments are conducted under the same reaction condition. For the transesterification of palm oil, the FAME percent is 77.96% , 59.30%, 58.76% and 69.44% when using KOH, CaO, CaCO3 and CaO/Zn catalyst respectively. As for the biodiesel produced from rubber seed oil, the FAME content percent is 80.02% , 66.61%, 63.92% and 75.82% when using KOH, CaO, CaCO3 and CaO/Zn catalyst respectively. The FAME content is the highest when using the KOH catalyst that is 77.96% when using palm oil and 80.02% when using rubber seed oil. The biodiesel produced when using CaO/ZnO catalyst has the nearest FAME content to the biodiesel produced when using KOH catalyst that is 69.44% when produced from palm oil and 75.82% when using rubber seed oil.

CHAPTER 5
CONCLUSION

5.1 Conclusion

The first objective of this project is to synthesize calcium-based catalyst and it was achieved. Catalysts used for this project are prepared by calcinations method for CaO and $CaCO_3$ and impregnation method for CaO/ZnO.

The second objective is to produce biodiesel through transesterification reaction by using the synthesized calcium-based catalyst. The product of the transesterification of RSO have been tested and the yield of fatty acid methyl ester are compared between biodiesel that produced using different catalyst. From Table 6, it can be seen that the homogeneous catalyst KOH had better performance than the heterogeneous catalyst CaO, $CaCO_3$ and CaO/ZnO. When comparing the FAME produce between the different type of oil but using same catalyst for transesterification, it can be observed that the biodiesel produced from rubber seed oil has more FAME content. When using palm oil to produce biodiesel with KOH as catalyst, the FAME content is about 77.96% whereas when using rubber seed oil, the FAME content is about 80.02%. The catalyst with highest performance among the calcium based catalyst is CaO/ZnO catalyst, this shows that the mixed calcium based catalyst has the potential to developed for the transesterification process.

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APPENDIX

Appendix A: Weight Loss Calculation

Catalyst	Time (hour)	Temperature (°C)	Weight before calcination (g)	Weight after calcination (g)
CaO	3	900	30.0078	17.1861
$CaCO_3$	3	600	60.1300	59.6000

Theoretical mass reduction

Mw $CaCO_3$: 100.08

Mw CaO: 56.08

Theoretical mass reduction for CaO

$$\text{Weight loss \%} = \left(\frac{100.08 - 56.08}{100.08}\right) \times 100$$

$$= 44\%$$

Actual mass reduction CaO

$$\text{Weight loss} = 30.0078 - 17.1861$$

$$= 12.8217 \text{ g}$$

$$\text{Weight loss \%} = \left(\frac{30.0078 - 12.8217}{30.0078}\right) \times 100$$

$$= 43\%$$

Theoretical mass reduction for $CaCO_3$

$$\text{Weight loss \%} = \frac{(100.08 - 100.08)}{100.08} \times 100$$

$$= 0\%$$

Actual mass reduction $CaCO_3$

$$\text{Weight loss} = 60.1300 - 59.6000$$

$$= 0.53 \text{ g}$$

$$\text{Weight loss \%} = \frac{(60.1300 - 0.53)}{60.1300} \times 100$$

$$= 0.88\%$$

Appendix B : Acidity Test.

Trial	Sample weight (g)	Volume of KOH used (ml)
1	1.0200	18.9
2	1.0230	19.2
3	1.0100	18.6

Free fatty acid can be calculated using formula:

$$FFA = \frac{MW \times vol \times N}{m \times f}$$

Where f is conversion value factors for RSO =2.81

First Trial

$$FFA = \frac{56.01 \times 18.9 \times 0.1}{1.0200 \times 2.81}$$

FFA = 36.99

Second Trial

$$FFA = \frac{56.01 \times 19.1 \times 0.1}{1.0230 \times 2.81}$$

FFA = 37.22

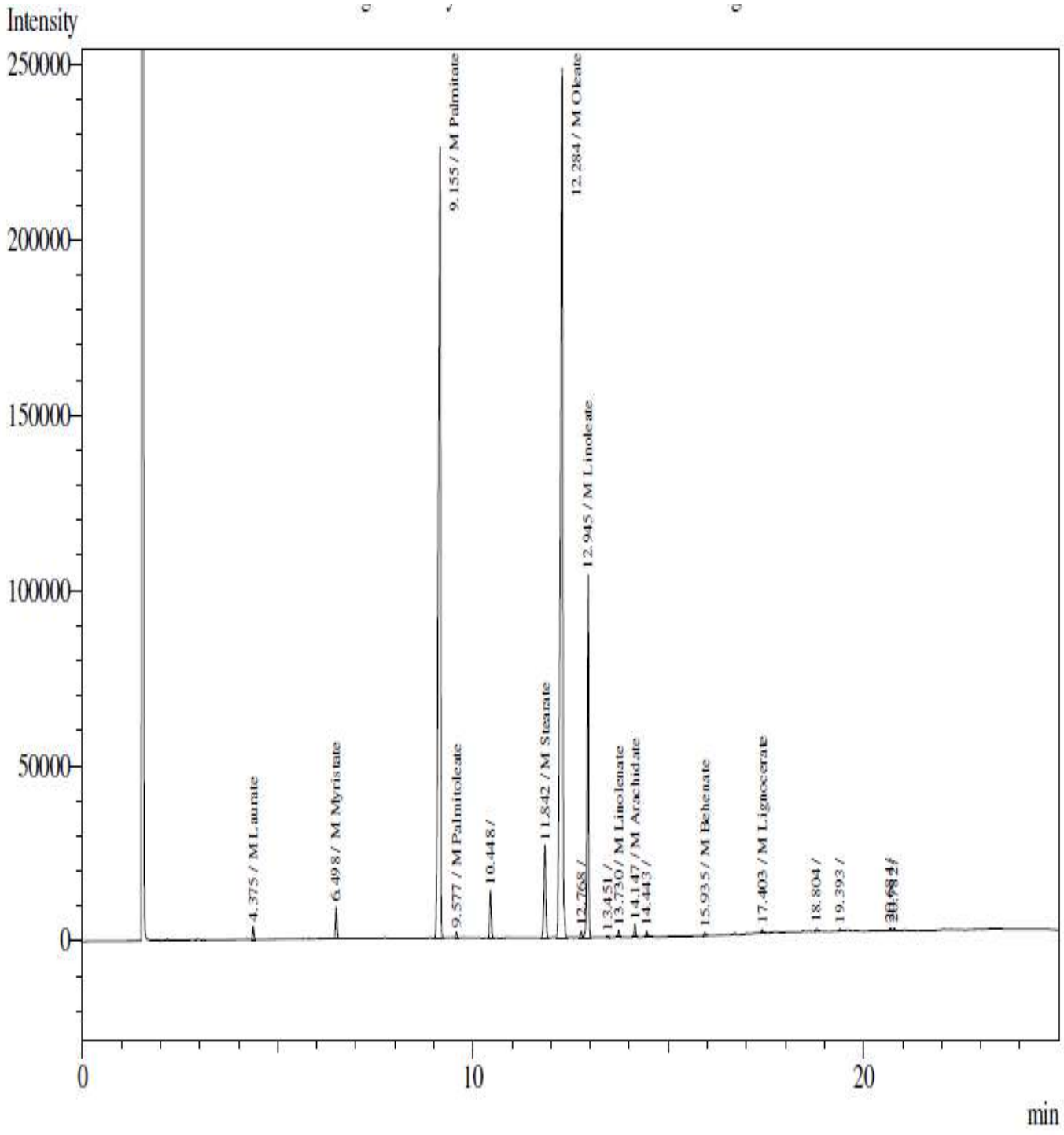
Third Trial

$$FFA = \frac{56.01 \times 18.6 \times 0.1}{1.0100 \times 2.81}$$

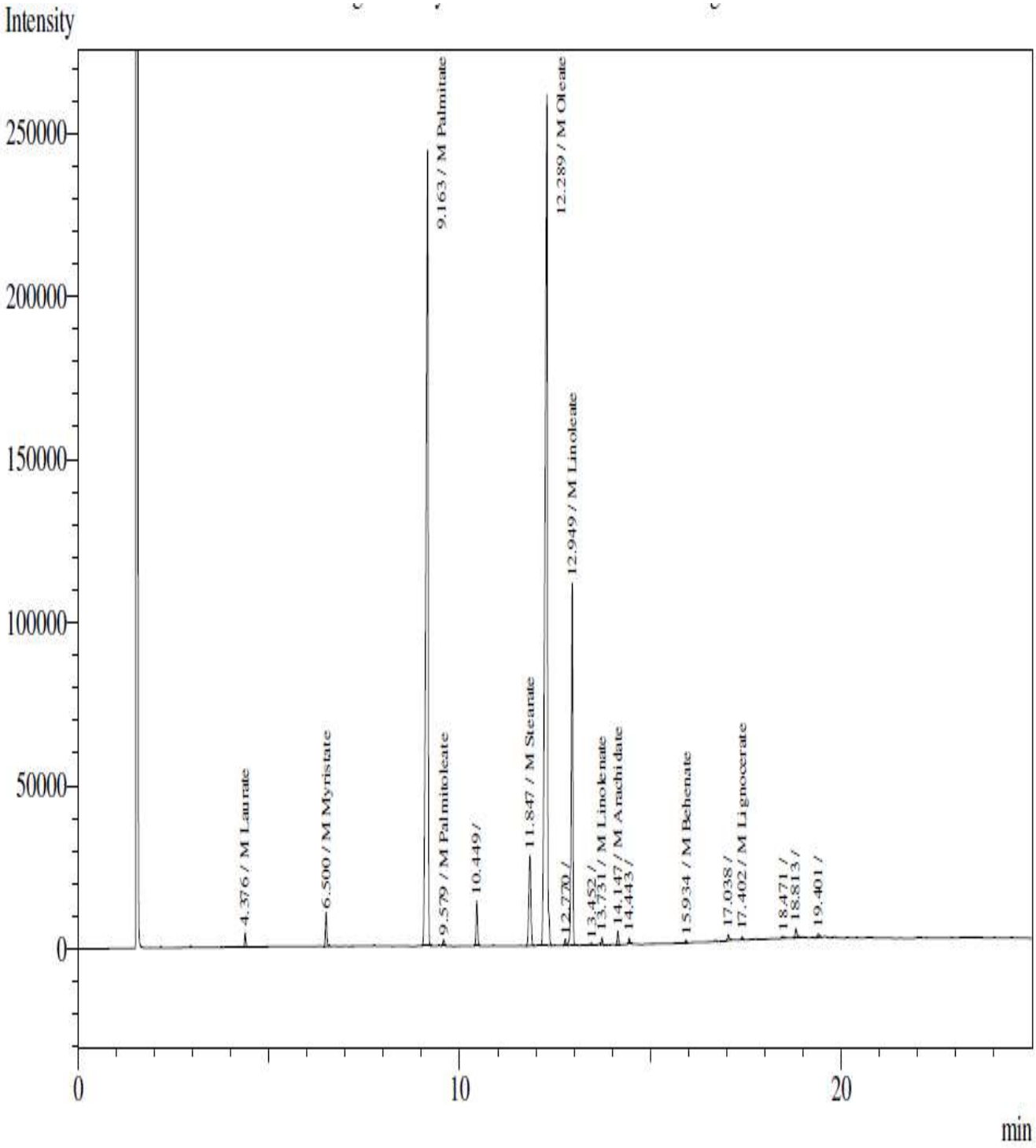
FFA = 36.77

Appendix C : GC Analysis Results

GC GRAPH FOR KOH CATALYST.



GC GRAPH FOR CaO CATALYST.



Oil Type	catalyst	Total peak area	MH peak area	concentration of MH (mg/ml)	MH volume (ml)	sample mass (mg)	FAME content (%)
Palm Oil	KOH	2400228	36580	0.625	1	51.8	77.96
Palm Oil	CaO	1975856	36565	0.625	1	55.9	59.30
Palm Oil	$CaCO_3$	1920056	36234	0.625	1	55.3	58.76
Palm Oil	CaO/Zn	1971237	34517	0.625	1	50.5	69.44
RSO	KOH	2642653	36545	0.625	1	51.7	86.21
RSO	CaO	1989135	36570	0.625	1	50.1	66.61
RSO	$CaCO_3$	1920985	36419	0.625	1	50.6	63.92
RSO	CaO/Zn	2245819	36284	0.625	1	50.2	75.82

$$C = \frac{\Sigma A - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100\%$$

Where:

$\sum A$ = total peak area $C_{14:0} - C_{24:1}$

A_{IS} = internal standard (methyl heptadecanoate) peak area

C_{IS} = concentration of the internal standard solution, in mg/mL

V_{IS} = volume of the internal standard solution used, mL

m = mass of the sample, in mg

SAMPLE CALCULATION

Transesterification of Palm Oil using KOH catalyst

$$C = \frac{(2400228 - 36580) \times 0.625 \times 1}{36580 \times 51.8}$$

C = 77.96

Transesterification of Palm Oil using KOH catalyst

$$C = \frac{(2655856 - 37565) \times 0.625 \times 1}{37565 \times 55.9}$$

$$C = 77.93$$